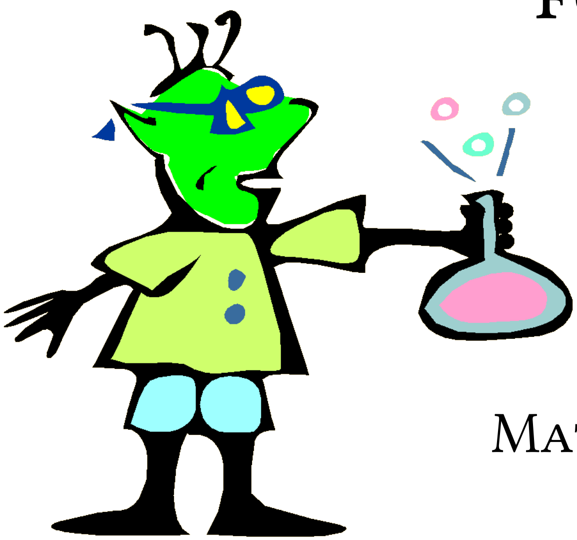
Optimizing Automated Particle Analysis for Forensic Applications



Nicholas W. M. Ritchie
Materials Measurement Science Division
Materials Measurement Lab
29-Nov-2012

The Big Picture

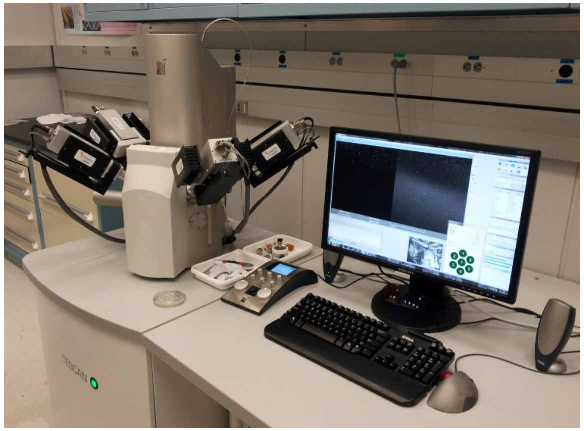
- X-ray Microanalysis
  - Electron Probe Microanalysis (EPMA)
  - Energy Dispersive X-ray Microanalysis (XEDS)
  - Microanalysis of challenging samples
    - Particles, fibers, films, inclusions, …
  - Microanalysis of particle data sets
- Customers
  - Material science, forensics, manufacturing,

Our Tools

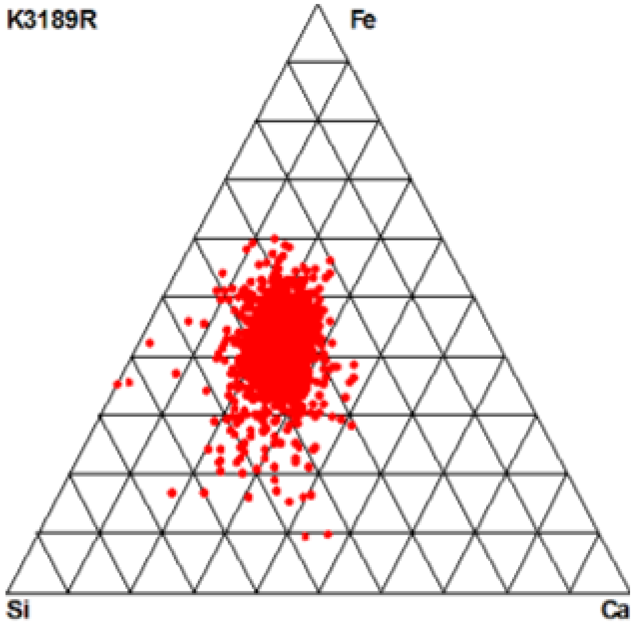
- Instruments
  - 2 electron microprobes, 2 FIBS, 2 FEG SEM, 1 W-filament SEM
- Software
  - NIST DTSA-II – Simulation & quantification
  - NIST Graf – Quantification, review & data mining of particle data sets
- Other techniques
  - STEM, XRF, EBSD, Atom Probe, Confocal, XRD, Auger

High Speed Automated Analysis of Particles using SEM/EDS

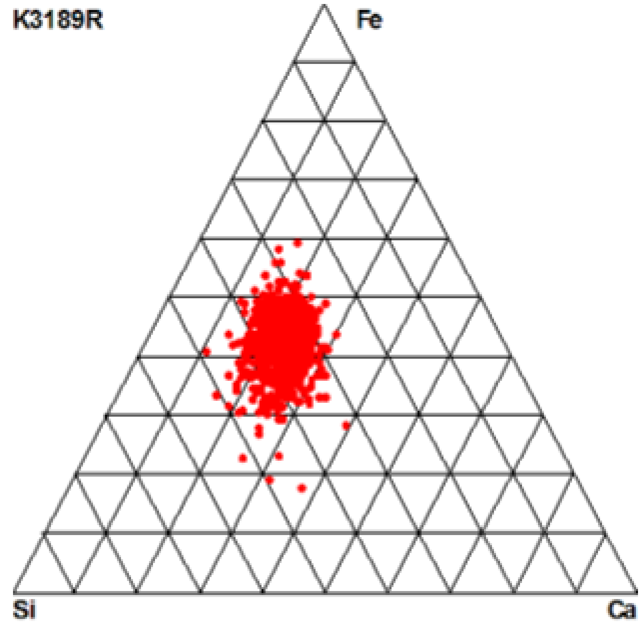
- High Speed – 10,000+ particle data sets
  - Moderate quality analyses of many particles
  - Search for a needle-in-a-haystack
- Automated – Configure, start then no operator intervention
  - Minimize operator bias
  - Reduce tedium
- Analysis -
  - Images and quantitative elemental analysis



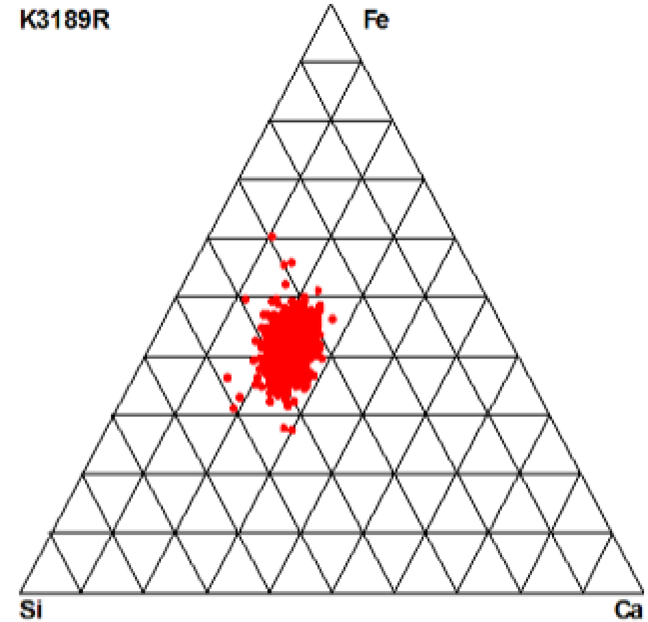
Conventional Si(Li) Detector – 6.4 μs process time



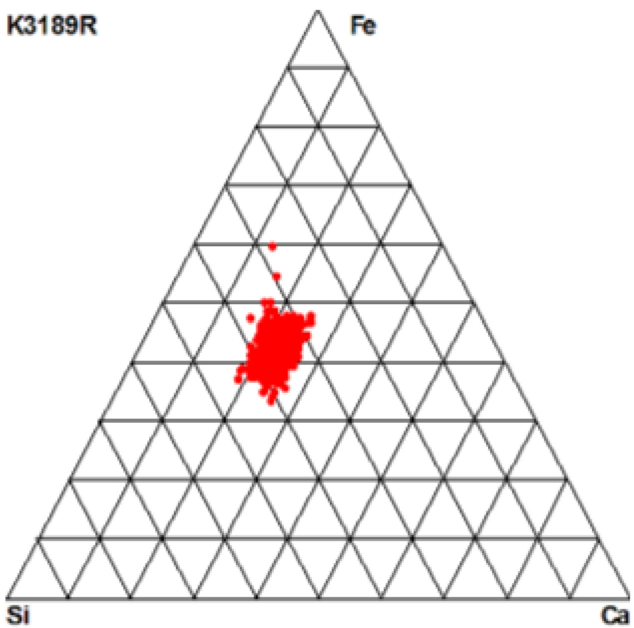
0.5 seconds



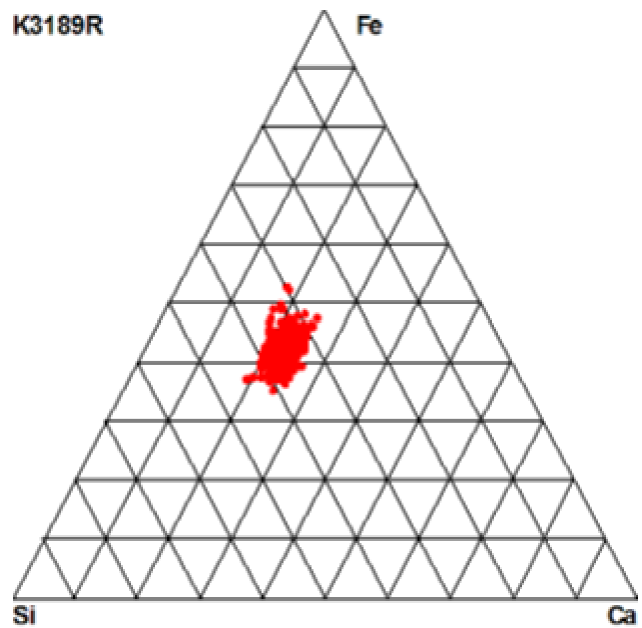
1.0 seconds



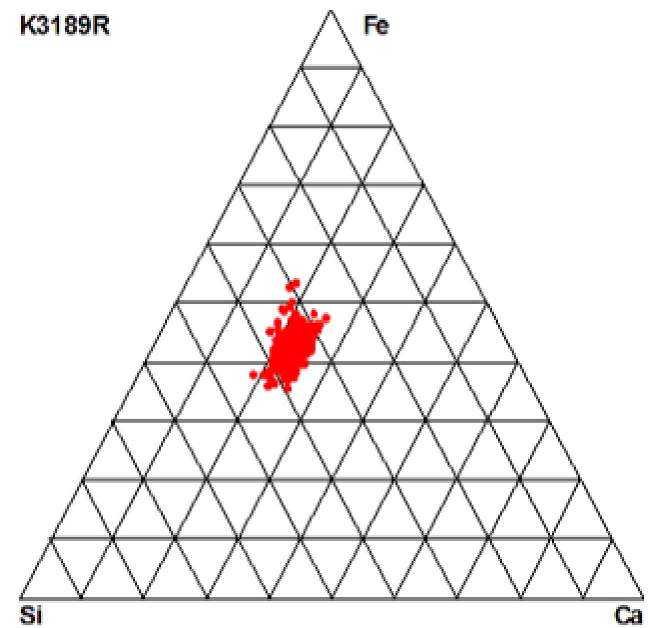
2.0 seconds



4.0 seconds



8.0 seconds



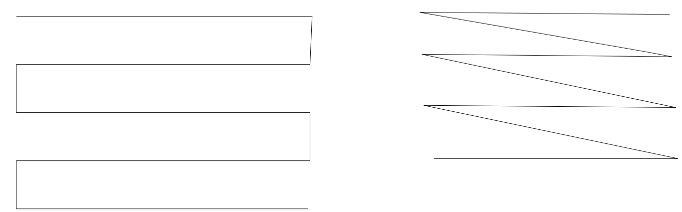
16.0 seconds

Major Time Sinks

- Stage motion – Tiling, stage speed
- Searching – Search pixel size, pixel dwell
- Measuring – Accuracy & pixel dwell
- Compositional Analysis – Limits-of-detection
- Mapping – Pixel dwell, area
- Overhead
  - QC

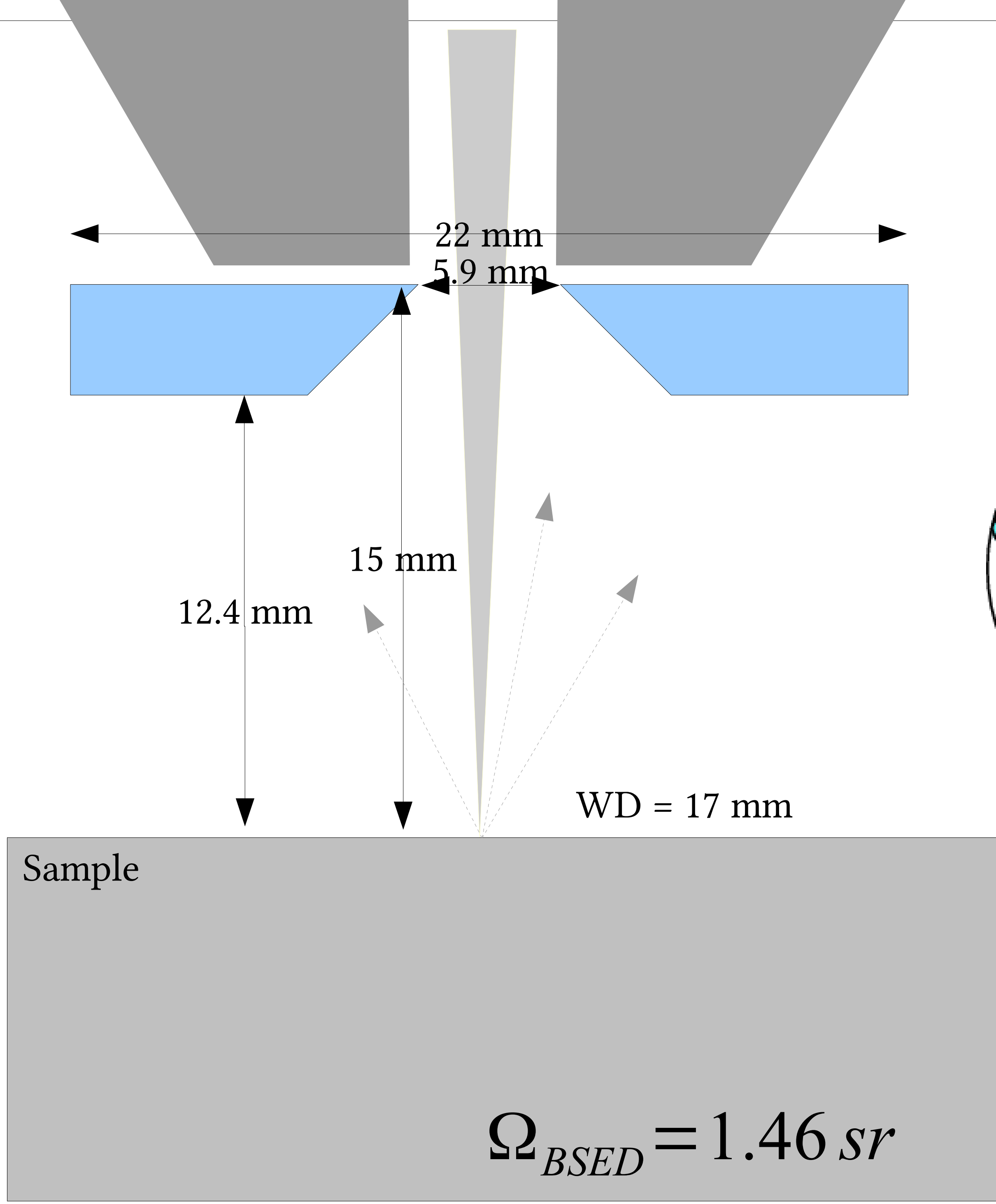
Strategies for Optimizing Stage Movement

- Speed up the stage
  - Particularly backlash removal jogs, post-move vibration
- Minimize stage movement
  - Move in serpentine
  - Subsets
    - Fixed size - Order frames to produce shortest path
    - Unknown size – Can't optimize path
  - Electronic fields – Move beam not stage



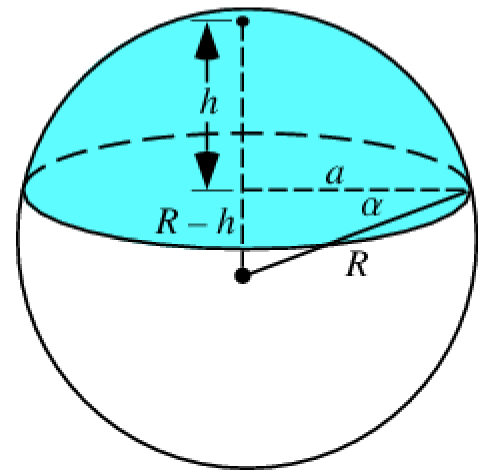
Optimizing the Backscatter Detector

- Consider a probe current of 1 nA and a dwell of 1 μs
  - $(1\ \text{nA})(1\ \mu\text{s}) = (6.241 \times 10^{18}\ e^{-}/\text{s})(10^{-9})(10^{-6}\ \text{s}) = 6{,}200\ e^{-}$
- Typical backscatter coefficients range from 5% to 50%
- If we could collect every electron from
  - $\Delta I/I = (3{,}100)^{1/2}/(3{,}100) = 1.8\%$
- We actually collect about 14%
  - $\Delta I/I = (430)^{1/2}/(430) = 4.8\%$
  - $\Delta I/I = (43)^{1/2}/(43) = 15\%$

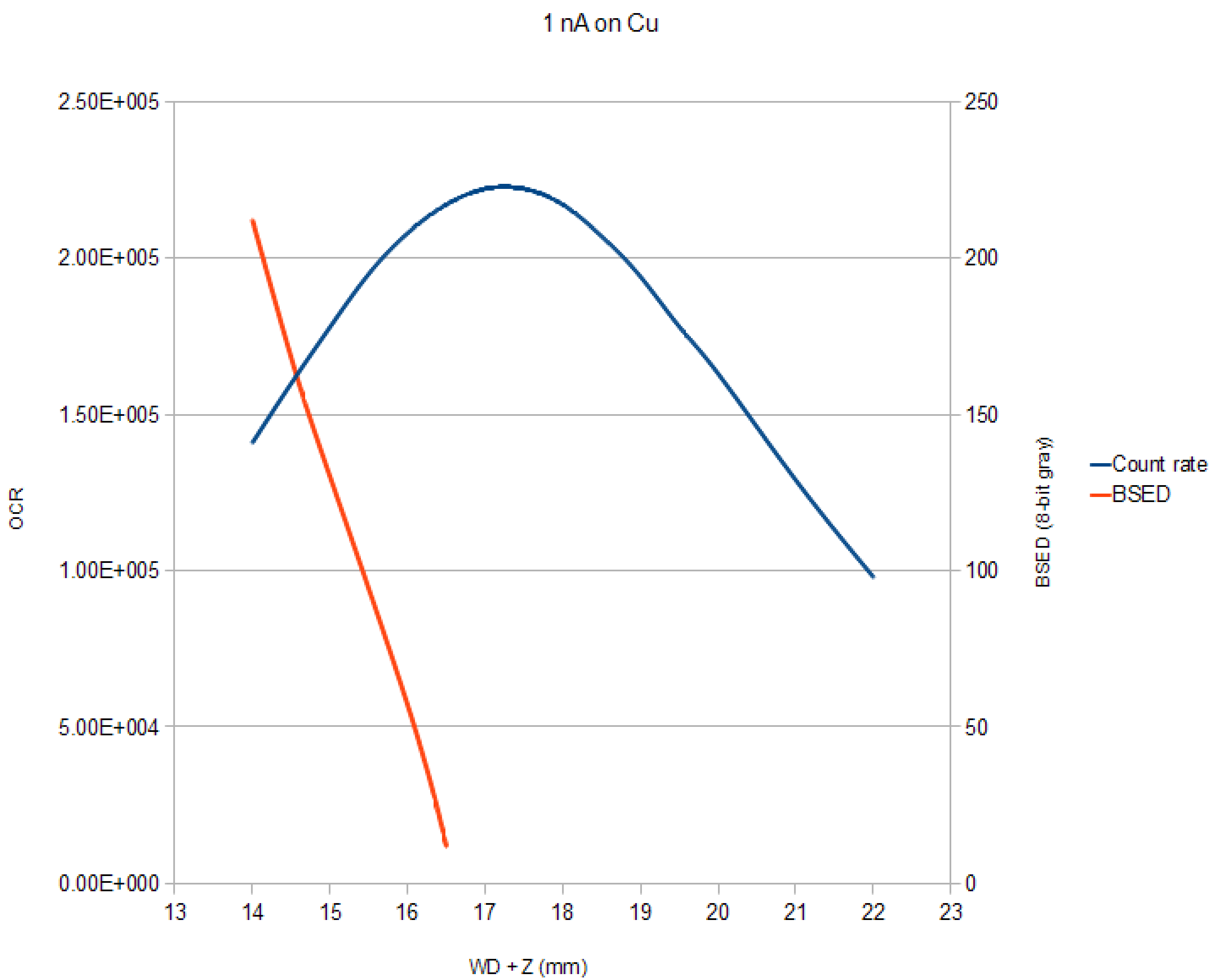


Area of a spherical cap

$$A = 2\pi R h$$

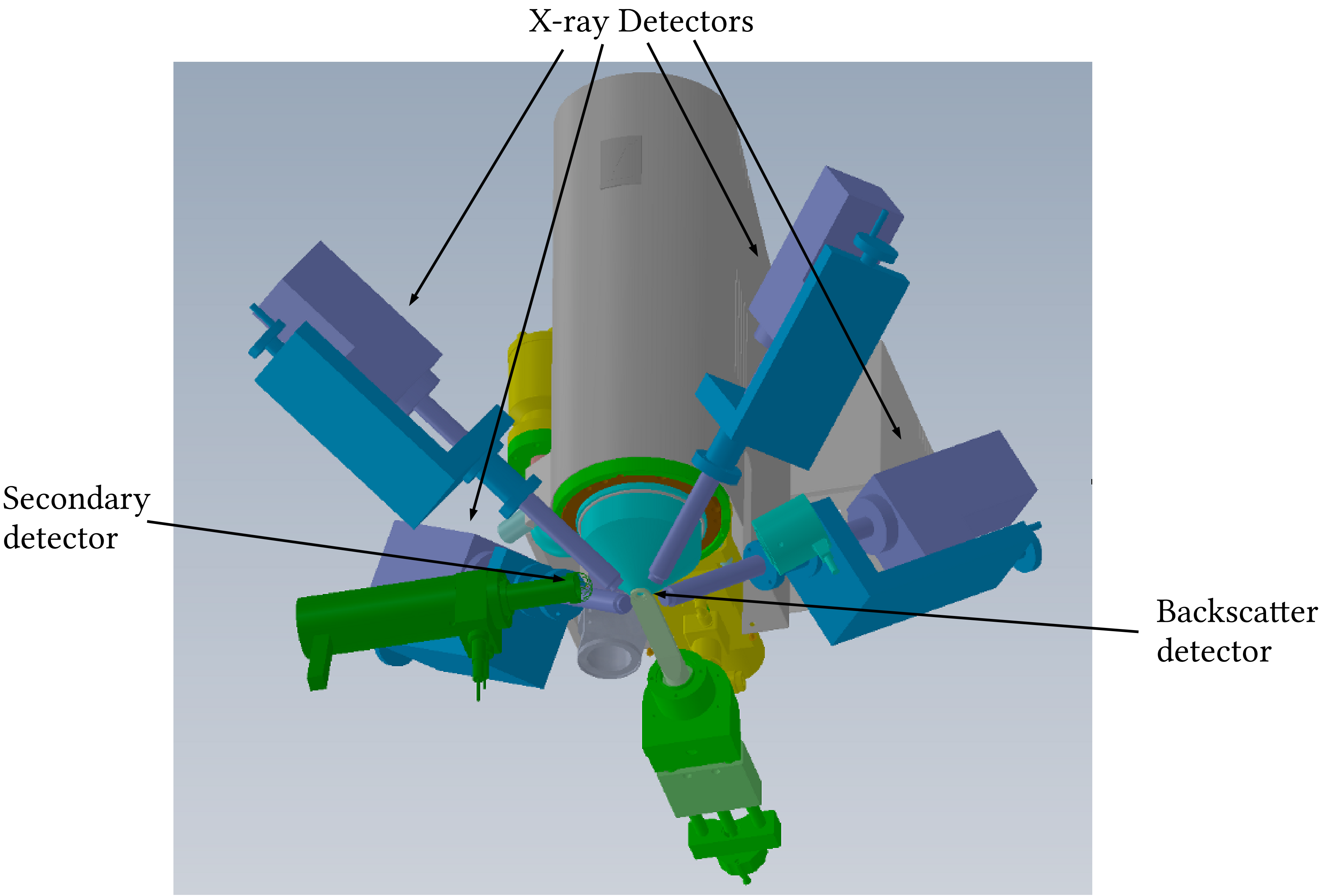


$$\Omega = \frac{2\pi R h}{R^2}$$

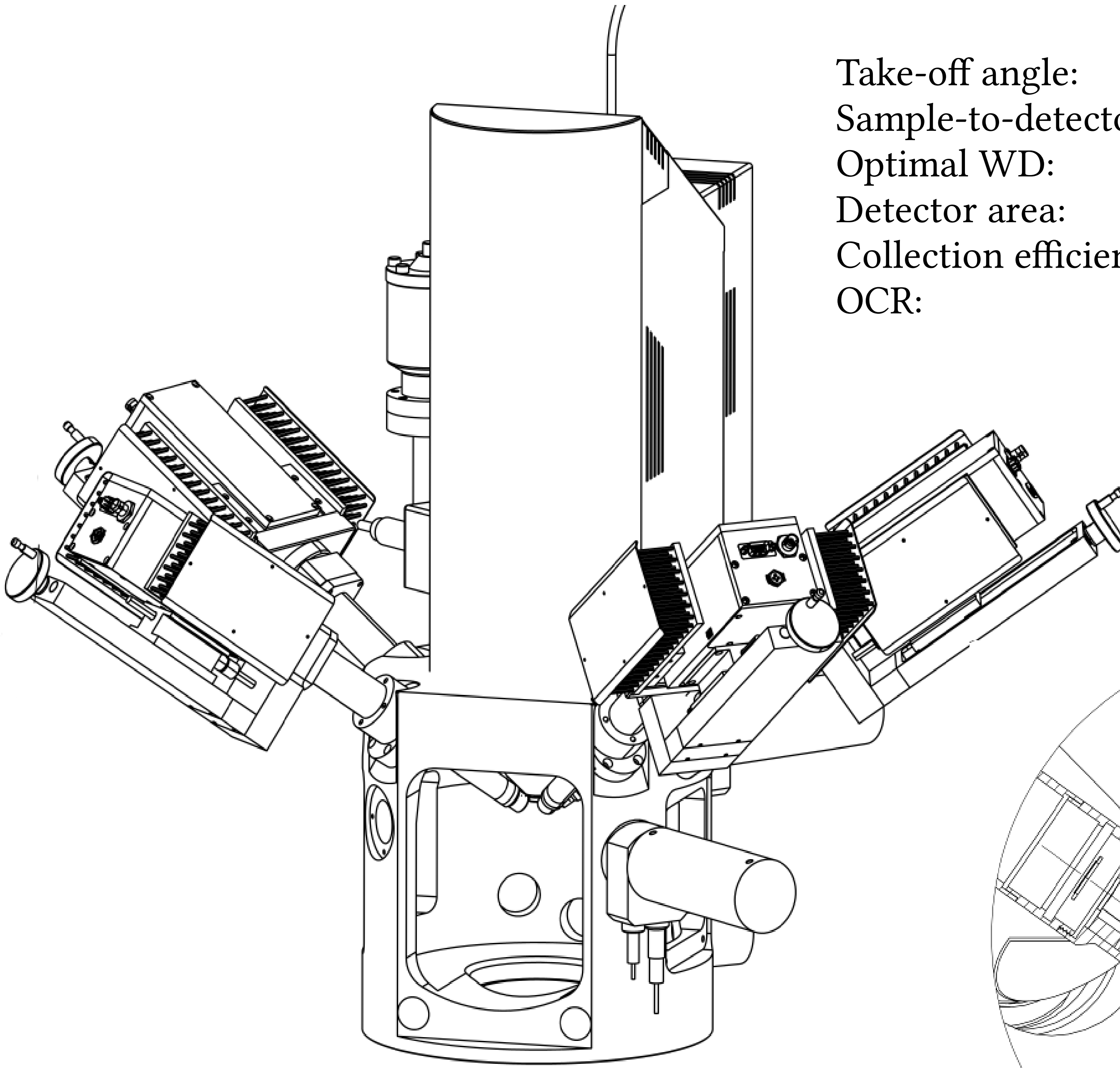

1 nA on Cu
2.50E+005
2.00E+005
1.50E+005
1.00E+005
5.00E+004
0.00E+000
OCR
250
200
150
100
50
0
BSED (8-bit gray)
13
14
15
16
17
18
19
20
21
22
23
WD + Z (mm)
Count rate
BSED

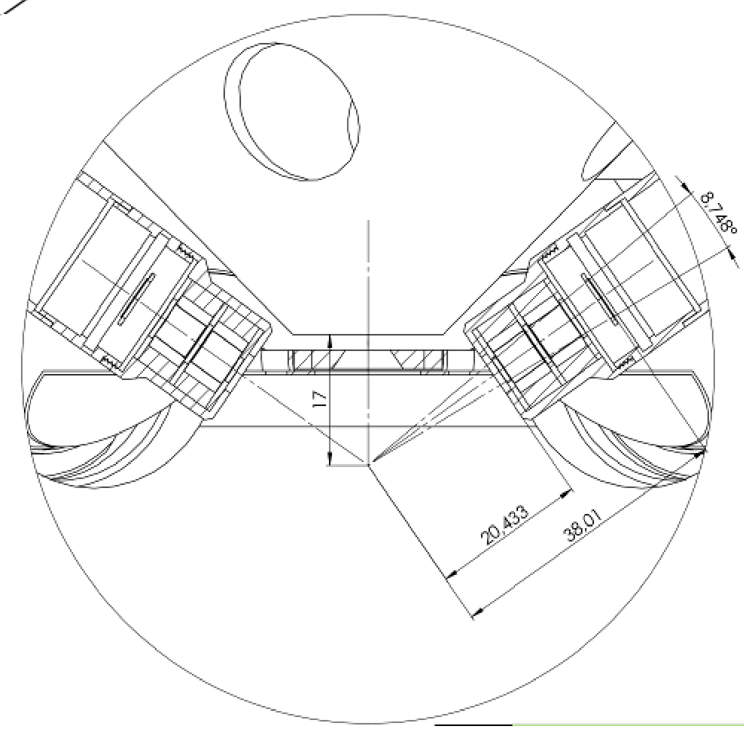
Optimizing EDS

- Maximize solid angle
  - Large area
  - In close
- Many angles better than one
  - Multiple detectors
- Many pulse processors better than one
  - Multiple pulse processors


X-ray Detectors
Secondary detector
Backscatter detector

Take-off angle:	35°
Sample-to-detector:	34 mm
Optimal WD:	17 mm
Detector area:	4 × 30 mm^2
Collection efficiency:	0.66%
OCR:	200 cps per nA on Cu

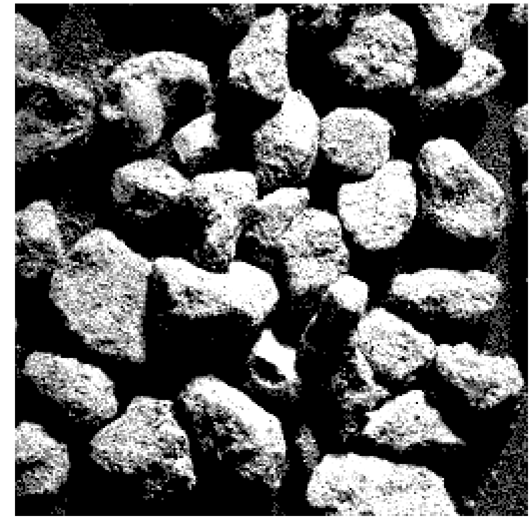




TESCAN MIRA3 with 4 PulseTor 30 mm^2 SDD

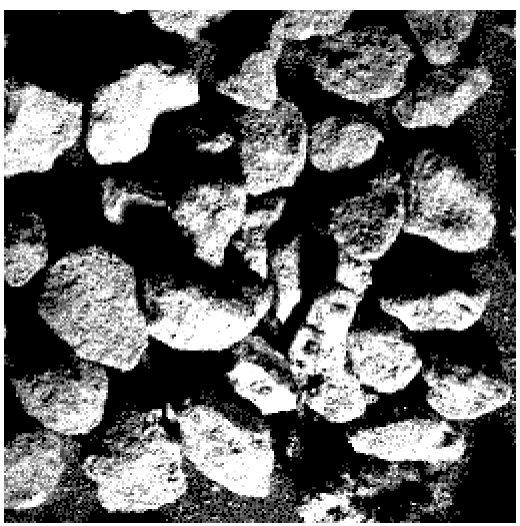
Oxygen in Iron Oxide Particles

Detector 2

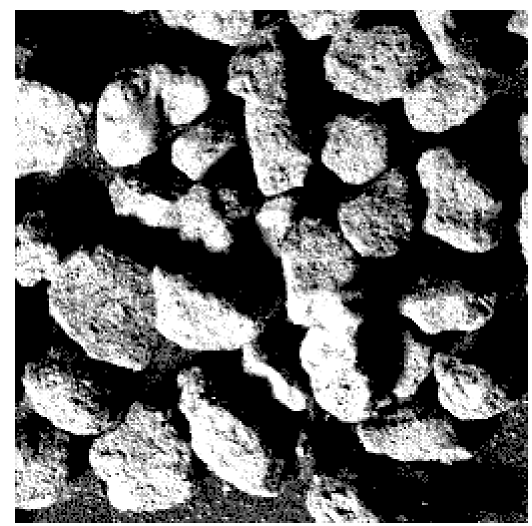


Detector 3

Sum



Detector 1

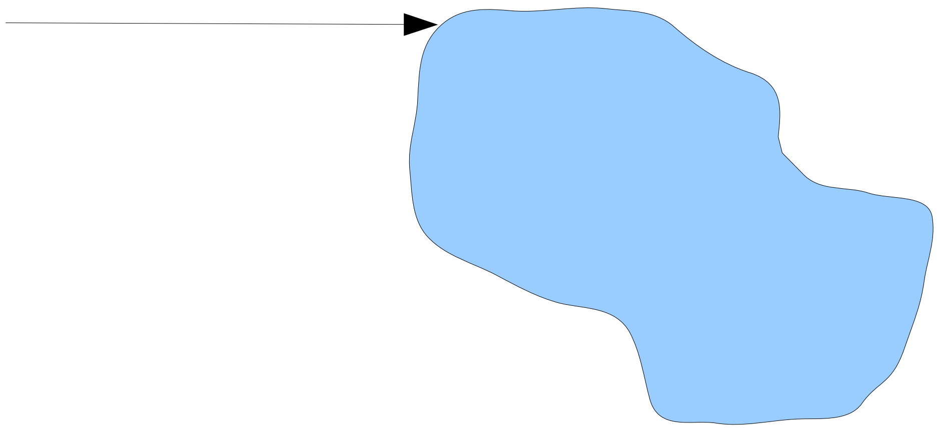


Detector 4

Sample: Raw iron oxide particles from Calvert Cliffs, MD

An SEM is not a Camera – Part 1

An SEM:
- Collects images pixel-by-pixel, row-by-row
- Can stop the raster anywhere
- Can change directions



- We can size a particle quickly regardless of whether it is large or small.
- The "coord-raster" can be used to keep the beam on the particle while collecting EDS.

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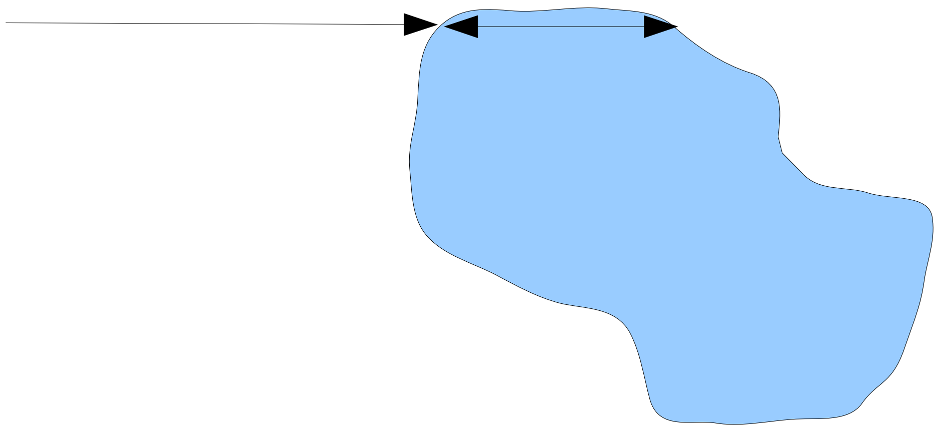

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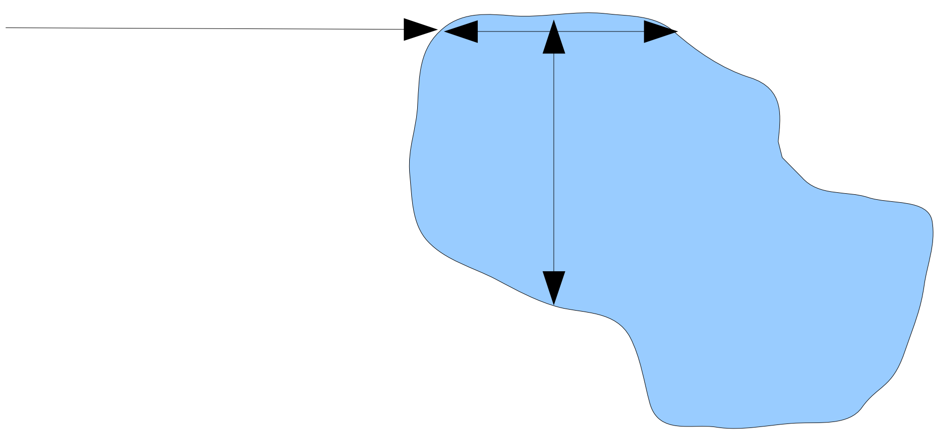

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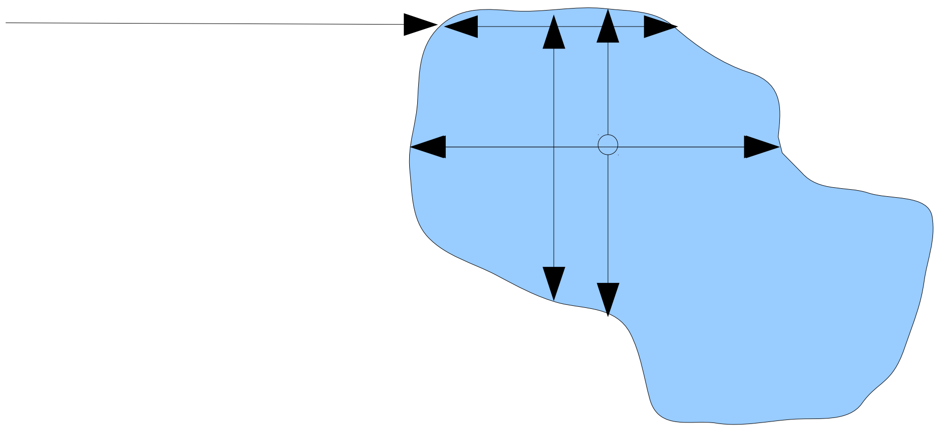

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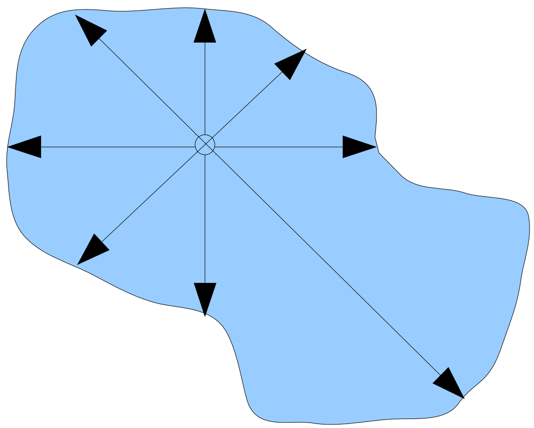

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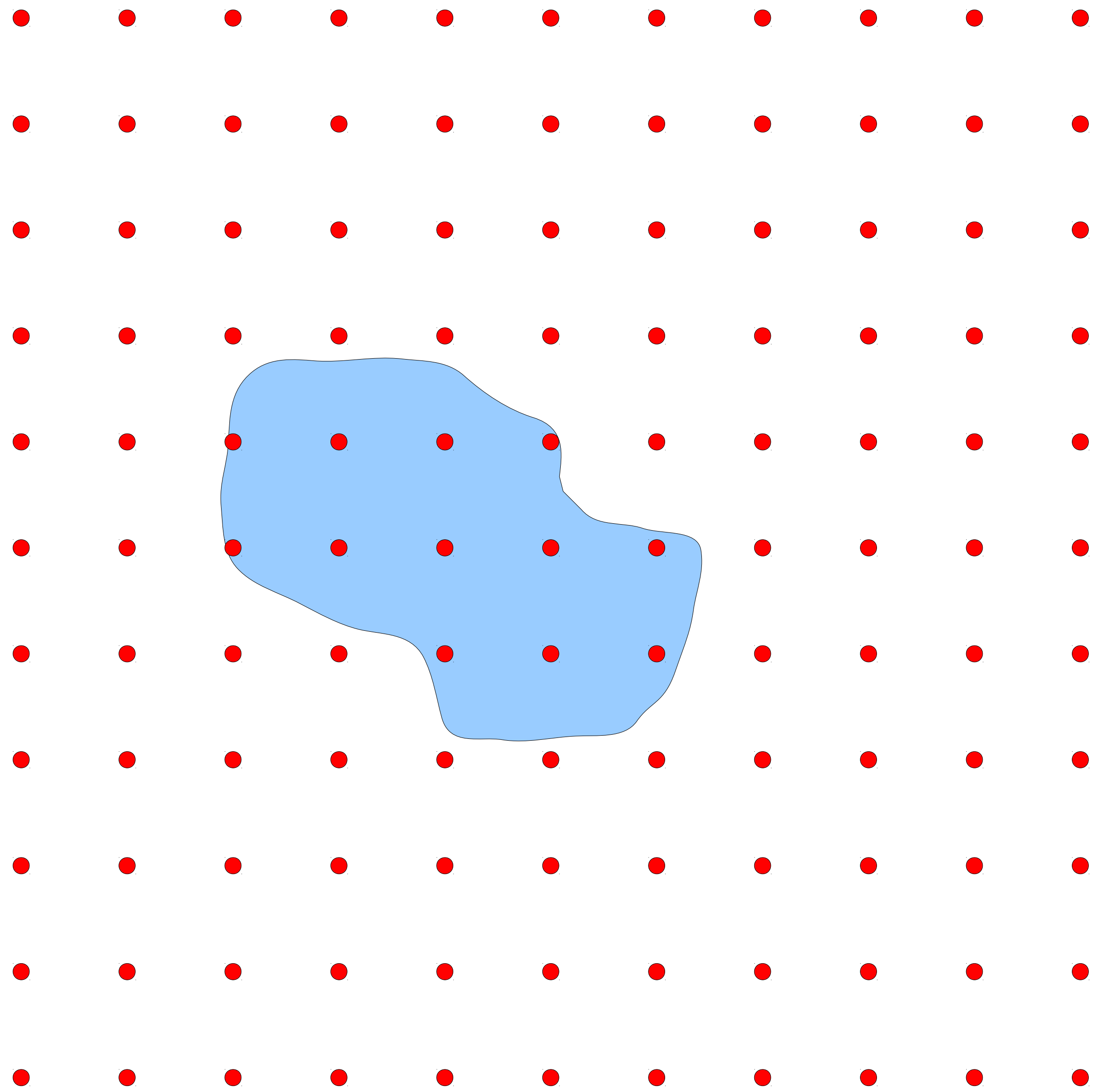
An SEM is not a Camera – Part 2

An SEM can:
- Dynamically change pixel spacing

- Search on a large pixel spacing
- Measure on a fine pixel spacing



Search
Measure

An SEM is not a Camera – Part 2

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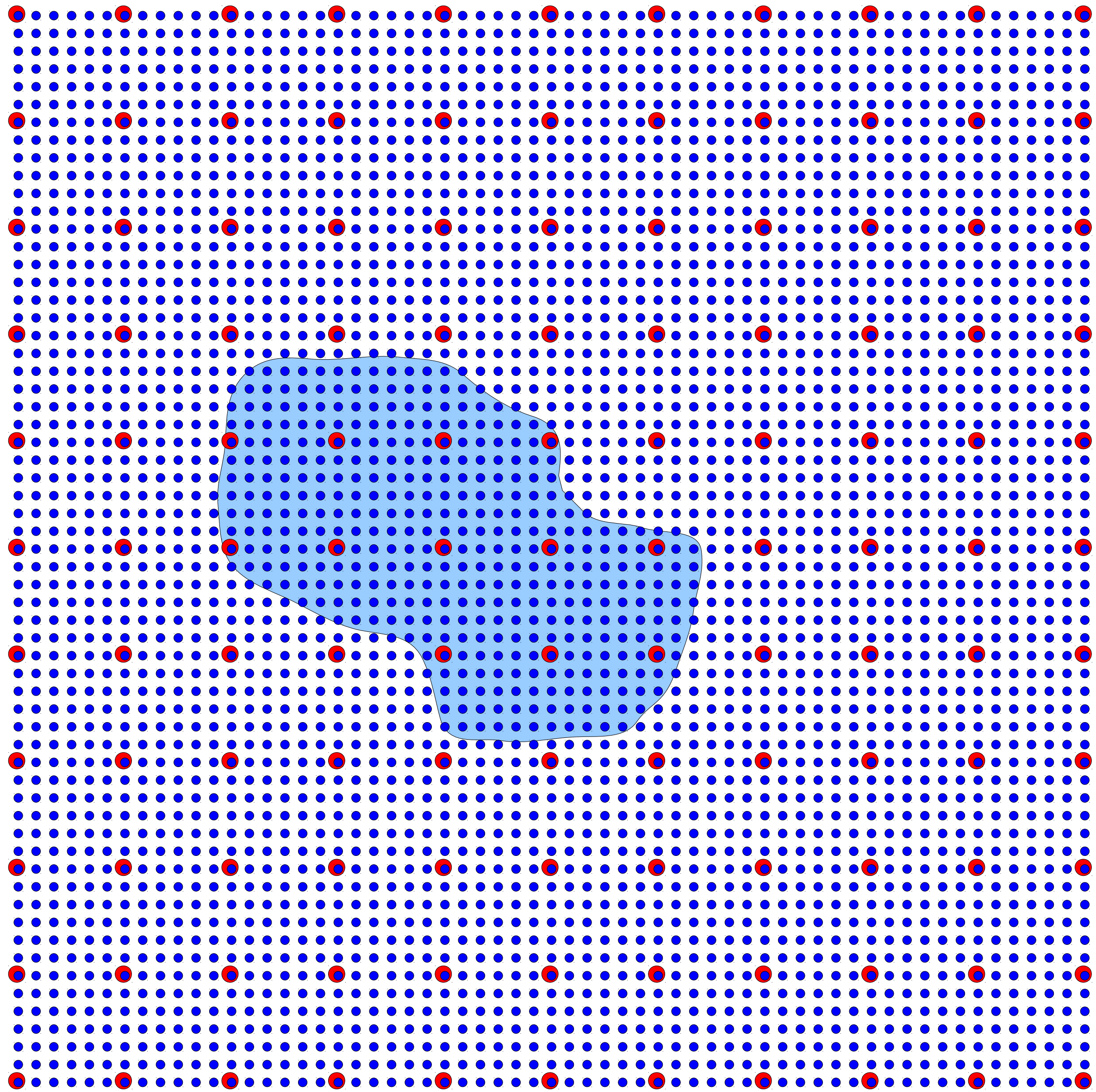


- Search on a large pixel spacing
- Measure on a fine pixel spacing

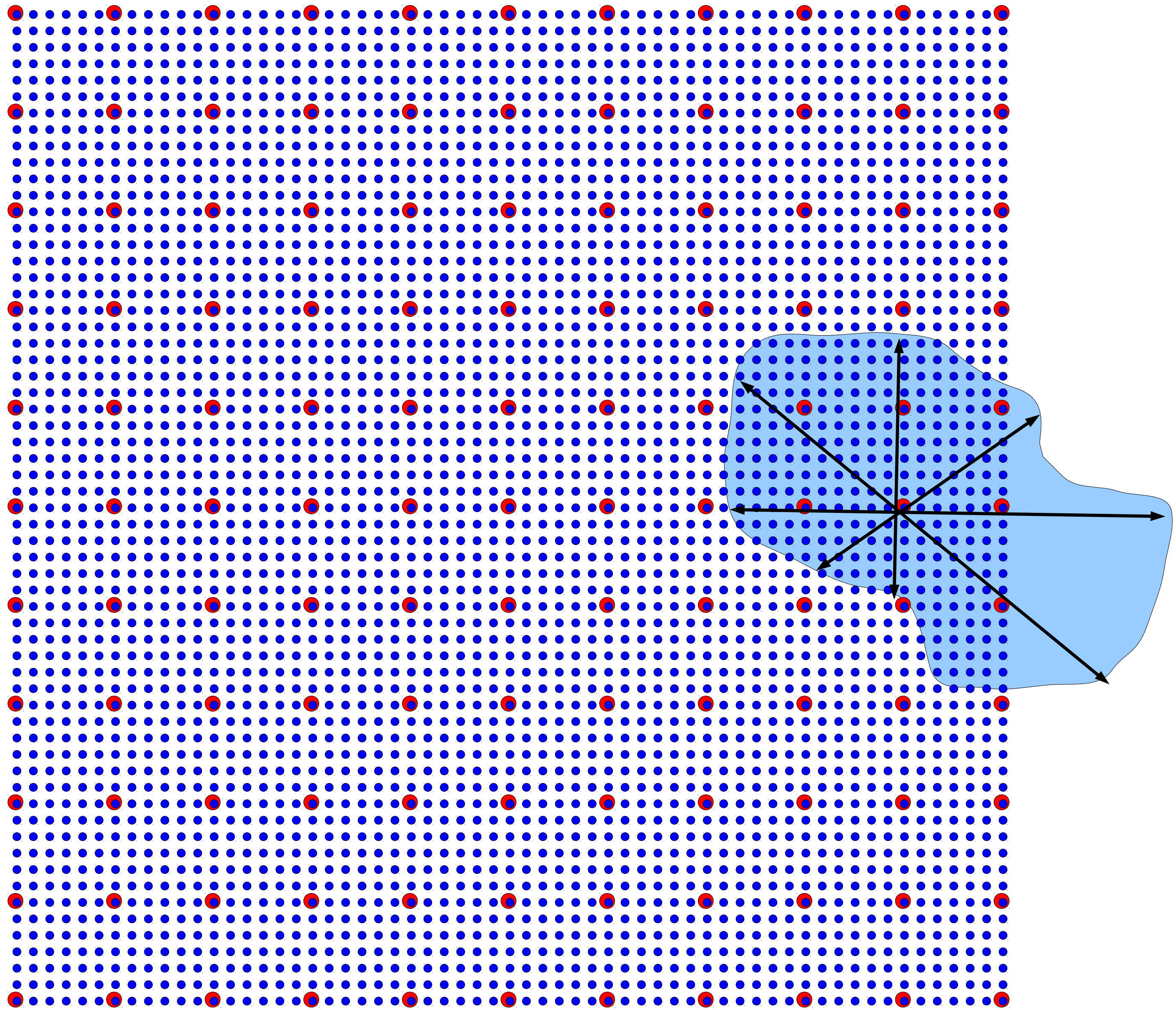
Search

Measure

An SEM is not a Camera – Part 3

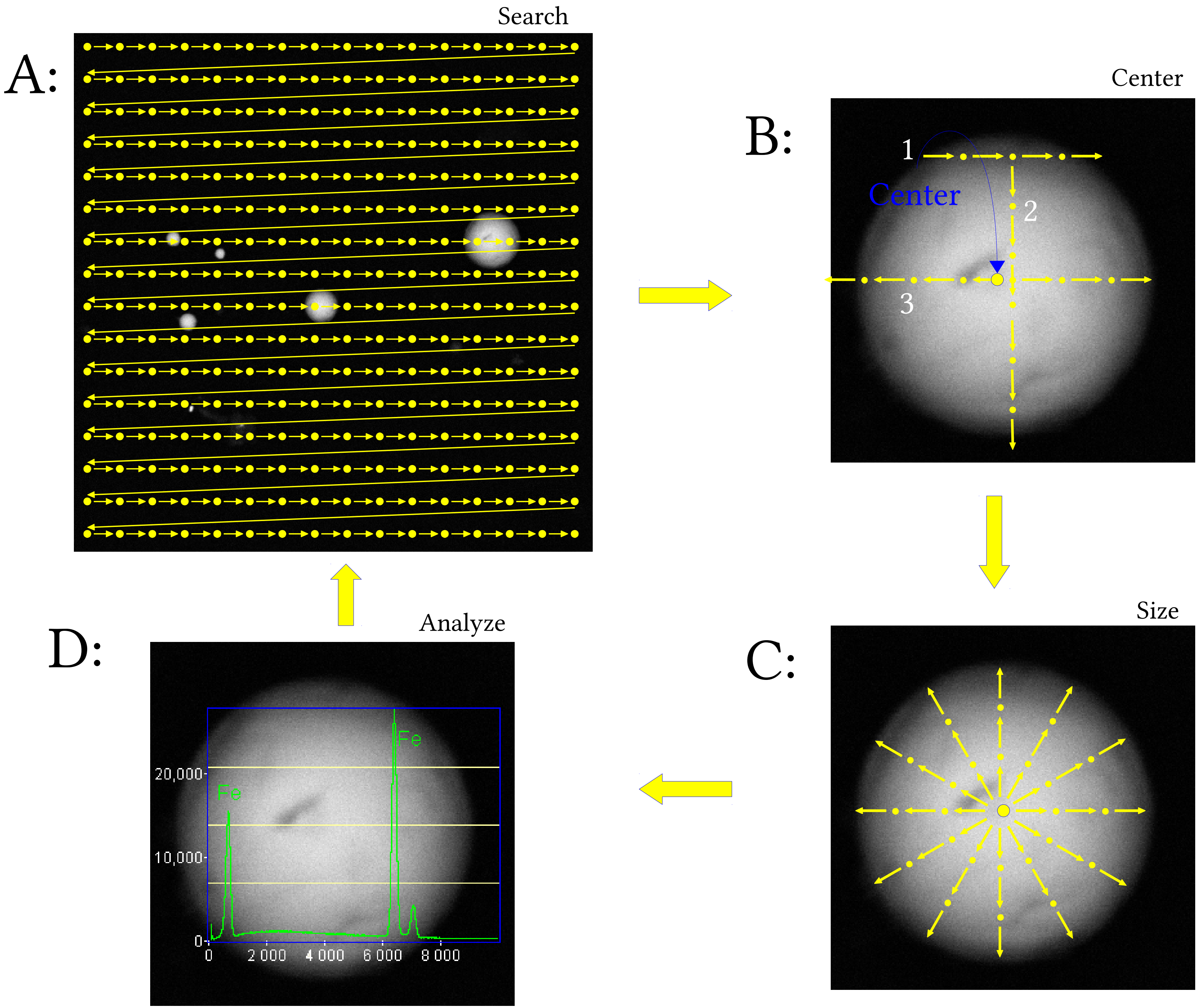
An SEM can:

- Raster the beam outside the nominal field-of-view
- Naturally handle particles that fall on a field edge.



Search

Measure



A:
Search
B:
Center
1
2
3
Center
C:
Size
D:
Analyze
20,000
10,000
0
Fe
Fe
0
2 000
4 000
6 000
8 000

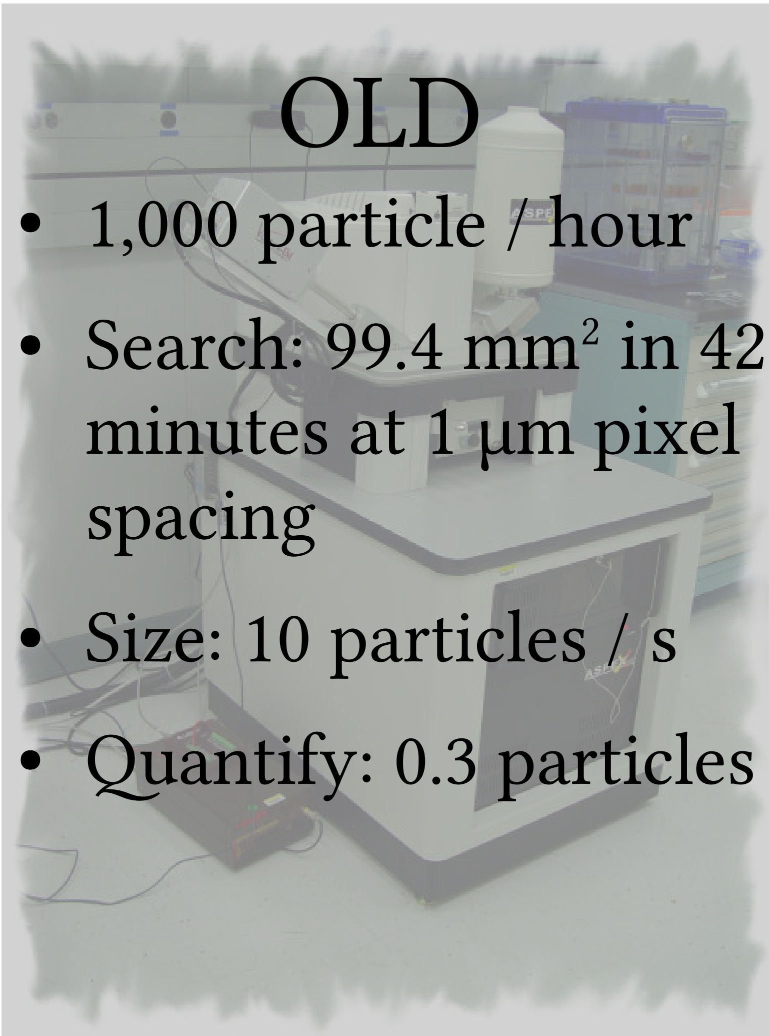
DOES IT MATTER?

	Dimensions		Timing	Precision		
	Search Pixels	*Measure* Pixels	*Search* (seconds)		*Overhead per particle* (seconds)	*Time for 100 particle field* (seconds)
Naive	2048	2048	4.194	1 part in 2048	0	4.19
Optimized	256	2048	0.066	1 part in 2048	~0.025	2.57

Optimized	Particles are sized and a spectrum collected as soon as discovered
Naive	Particles are sized and spectrum collected at the end-of-frame

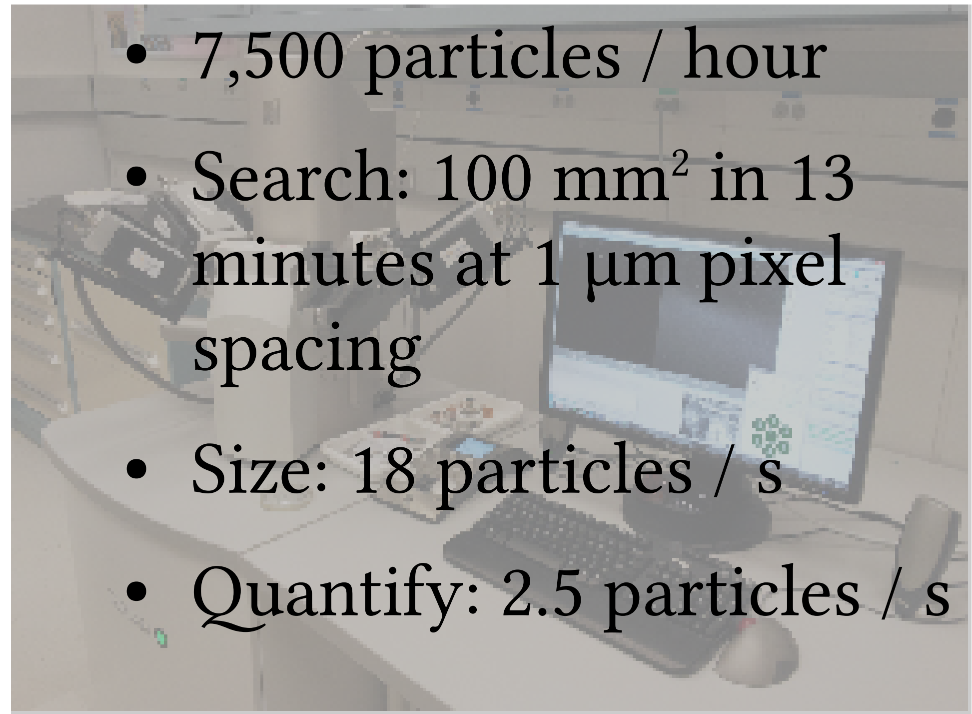
Fewer small particles are lost during analysis using the optimized algorithm.

Does it work?



OLD

- 1,000 particle / hour
- Search: 99.4 mm^2 in 42 minutes at 1 µm pixel spacing
- Size: 10 particles / s
- Quantify: 0.3 particles / s



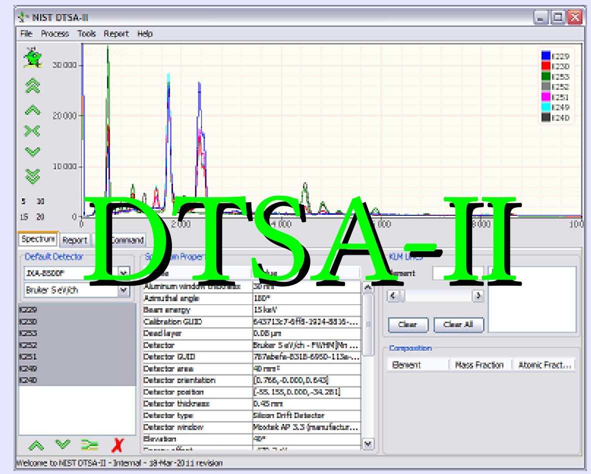
NEW

- 7,500 particles / hour
- Search: 100 mm^2 in 13 minutes at 1 µm pixel spacing
- Size: 18 particles / s
- Quantify: 2.5 particles / s

Tying it all together

SharkSEM API

NIST's contribution



SEMantics

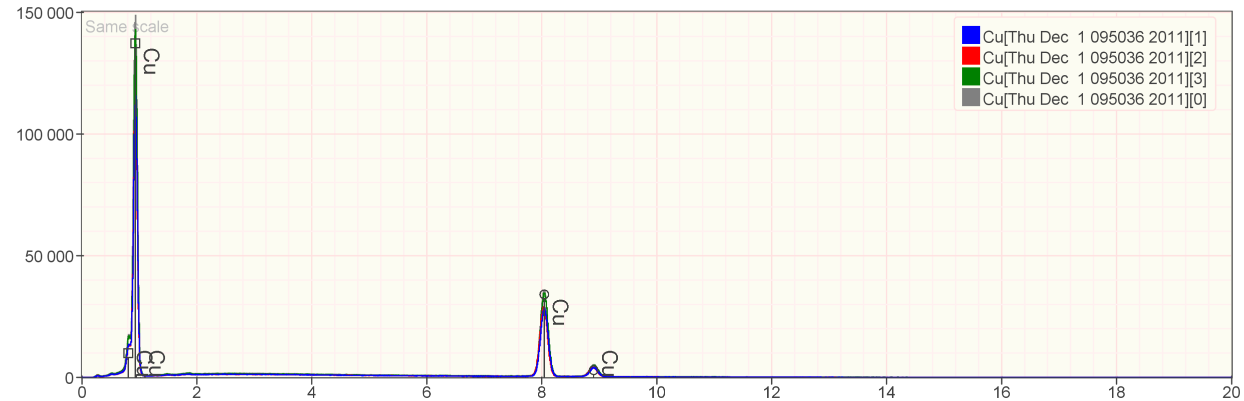
EM Automation Library in Java

+ python™ +

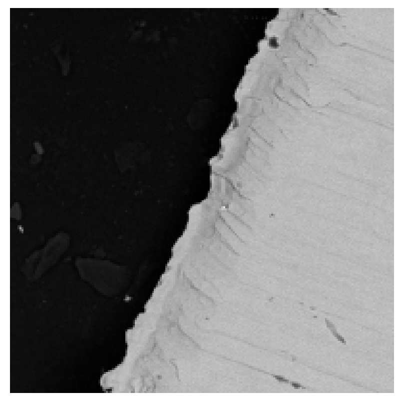
PostgreSQL The world's most advanced open source database.

A Final Word on QC

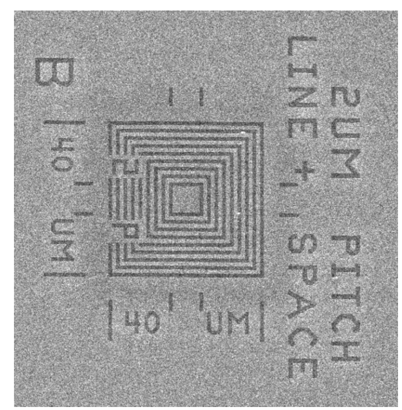
1) EDS



2) Imaging detectors



3) Magnification



4) Probe current